

## PATENT ABSTRACTS OF JAPAN

(11)Publication number : 05-148768

(43)Date of publication of application : 15.06.1993

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(51)Int.Cl.

D06M 15/53  
D06M 13/322  
// D06M101:16

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(21)Application number : 03-340309

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(22)Date of filing : 28.11.1991

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### (54) METHOD FOR GIVING HYDROPHILICITY TO SURFACE OF HYDROPHOBIC SYNTHETIC FIBER

(57)Abstract:

PURPOSE: To give excellent durability and high hydrophilicity to the surface of a fiber by compounding a specific polyether-ester compound and/or a polyether-amide compound with a specified nitrogen-containing compound and adding a specific amount of the mixture to the synthetic fiber.

CONSTITUTION: (A) A polyether-ester compound and/or a polyether-amide compound is compounded with (B) a nitrogen-containing compound containing one or more ammonium groups and two or more  $\geq 12$ C hydrocarbon groups in the molecule as a nitrogen-containing compound in an A/B weight ratio of 95/5 to 50/50, and the mixture is dispersed in water. A synthetic fiber is dipped in the produced aqueous dispersion, and the dispersion is stuck to the fiber in an amount of 0.1-3%owf to give high initial water dispersibility and excellent hydrophilicity to the fiber. The component A includes polyetherester compounds produced from saturated aliphatic dicarboxylic acid and a glycol, etc., and the amide includes a compound produced from a dicarboxylic acid, a diamine and a polyetherdiol, etc. The treating agent gives remarkable effects to polyester fibers.

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### LEGAL STATUS

[Date of request for examination]

01.12.1997

[Date of sending the examiner's decision of

rejection]

[Kind of final disposal of application other than  
the examiner's decision of rejection or  
application converted registration]

[Date of final disposal for application]

[Patent number] 2999616

[Date of registration] 05.11.1999

[Number of appeal against examiner's  
decision of rejection]

[Date of requesting appeal against examiner's  
decision of rejection]

[Date of extinction of right]

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CLAIMS

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[Claim(s)]

[Claim 1] The hydrophilic graft approach to the hydrophobic synthetic-fiber front face characterized by being the rate of A/B=95 / 5 - 50/50 (weight ratio), and using the polyether ester of Following A and/or polyether ester amides, and the nitrogen-containing compound of Following B so that such sum total coating weight may become 0.1 - 3 % of the weight to a hydrophobic synthetic fiber, and processing a hydrophobic synthetic fiber.

A: the following -- the dicarboxylic acid of one sort chosen from the polyether ester of A-1, and the polyether ester amides of following A-2, more than 2 sort A-1; aromatic series, or saturation aliphatic series -- more than 85 mol % -- with the included dicarboxylic acid or its ester plasticity derivative, and a glycol the dicarboxylic acid of the polyether ester A-2; aromatic series obtained from polyether diol, or saturation aliphatic series -- more than 90 mol % -- with the included dicarboxylic acid or its ester plasticity derivative The amide plasticity compound chosen from diamine, a lactam, and an amino carboxylic acid, The polyether ester amides B obtained from polyether diol: One sort or two sorts or more which are chosen as intramolecular from the nitrogen-containing compound which has one or more ammonium and the hydrocarbon group of two or more carbon numbers [ 12 or more ]

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DETAILED DESCRIPTION

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[Detailed Description of the Invention]

[0001]

[Industrial Application] This invention relates to the hydrophilic grant approach to a hydrophobic synthetic-fiber front face. Hydrophobic synthetic fibers including polyester fiber have the outstanding physical property and the profitability which was excellent in addition to the chemistry property, therefore are widely used for the object for garments, the object for bedclothing, industrial use, etc. from before. And in recent years, application development in which the profitableness was harnessed is performed positively, and the application application as which having the hydrophilic property excellent in front faces including fiber, such as health material and paper making, is required has been increasing as one of them. Especially, in manufacture of the wet nonwoven fabric represented by the paper-making application, it is the relation between quality improvement of a product, improvement in the speed of a paper-making process, etc., and an outstanding hydrophilic property which distributes to a single fiber promptly when fiber is supplied underwater and by which stable maintenance of it is carried out is required increasingly. This invention relates to the hydrophilic grant approach to the hydrophobic synthetic-fiber front face which meets this demand.

[0002]

[Description of the Prior Art] Conventionally, the following methods of processing a hydrophobic synthetic-fiber front face as the hydrophilic grant approach to a hydrophobic synthetic-fiber front face using various kinds of hydrophilic compounds are proposed. For example, there are an example (JP,45-10794,B, JP,60-134071,A, USP4410687) using the hydrophilic polyester which introduced the hydrophilic radical of 1 various kinds about polyester fiber, an example (JP,57-112411,A, JP,60-146081,A) which uses together 2 hydrophilic-property polyester and other water soluble polymers, an example (JP,61-12978,A, JP,62-162077,A) which uses together 3 hydrophilic-property polyester and a reactant compound, an example (JP,47-2512,B, JP,51-26400,A) which uses together 4 hydrophilic-property polyester and an anion mold surfactant. Moreover, especially about the polyester fiber for wet nonwoven fabrics, there are an example (JP,58-208500,A) using the polyether ester which has 1 polyalkylene glycol ether radical, an example (JP,1-298297,A) using the hydrophilic polyester which has 2 sulfonic groups, an example (JP,56-169814,A) using 3 polyether polyol, etc. Further for example, there is an example (JP,60-81052,A) which uses poly alkylamino acrylate about an acrylic fiber.

[0003] However, these conventional methods have the fault that the endurance is inferior, even if grant of a hydrophilic property is inadequate from the first or it can give a suitable hydrophilic property at the beginning. In a conventional method, if these are applied to manufacture of for example, a wet nonwoven fabric, when the hydrophobic synthetic fiber which carried out surface treatment is thrown in underwater, it will not distribute to a single fiber promptly, or stable maintenance of this distributed condition will not be carried out.

[0004]

[Problem(s) to be Solved by the Invention] Even if the conventional method of grant of a hydrophilic property is [ the technical problem which this invention tends to solve ] inadequate from the first or it

can give a suitable hydrophilic property at the beginning, it is a point inferior to the endurance.

[0005]

[Means for Solving the Problem] The deer was carried out, this invention persons are predetermined rates, respectively, and as a result of inquiring in order to solve the above-mentioned technical problem, they used specific polyether ester and/or polyether ester amides, and a specific nitrogen-containing compound so that such sum total coating weight might turn into the specified quantity, and the method of processing a hydrophobic synthetic fiber found out the surely suitable thing.

[0006] That is, this invention is the rate of A/B=95 / 5 - 50/50 (weight ratio), and the polyether ester of Following A and/or polyether ester amides, and the nitrogen-containing compound of Following B are used for it so that such sum total coating weight may become 0.1 - 3 % of the weight to a hydrophobic synthetic fiber, and it relates to the hydrophilic grant approach to the hydrophobic synthetic-fiber front face characterized by processing a hydrophobic synthetic fiber.

[0007] A: the following -- the dicarboxylic acid of one sort chosen from the polyether ester of A-1, and the polyether ester amides of following A-2, more than 2 sort A-1; aromatic series, or saturation aliphatic series -- more than 85 mol % -- with the included dicarboxylic acid or its ester plasticity derivative, and a glycol the dicarboxylic acid of the polyether ester A-2; aromatic series obtained from polyether diol, or saturation aliphatic series -- more than 90 mol % -- with the included dicarboxylic acid or its ester plasticity derivative The polyether ester amides [0008] obtained from the amide plasticity compound chosen from diamine, a lactam, and an amino carboxylic acid, and polyether diol B: One sort or two sorts or more which are chosen as intramolecular from the nitrogen-containing compound which has one or more ammonium and the hydrocarbon group of two or more carbon numbers [ 12 or more ]

[0009] As dicarboxylic acid of the aromatic series which will constitute the polyether ester of A-1, or saturation aliphatic series, saturation aliphatic series dicarboxylic acid, such as aromatic series dicarboxylic acid, such as 1 terephthalic-acid, isophthalic acid, orthochromatic phthalic-acid, 2, and 6-naphthalene dicarboxylic acid, 2 adipic acids, an azelaic acid, and a sebacic acid, is mentioned.

[0010] As a glycol which will constitute the polyether ester of A-1 1) Ethylene glycol, 1, 2-propylene glycol, 1,4-butanediol, Aliphatic series glycols, such as neopentyl glycol and 1,6-hexanediol, 2) Alicycle group glycols, such as a cyclohexane dimethanol, 2, and 2'-bis(4-hydroxy cyclohexyl) propane, 3) 2 and 2'-{bis(4-hydroxy ethoxy phenyl)} propane, Aliphatic series ether glycols, such as a glycol containing aromatic series radicals, such as 2 and 2'-{bis(4-hydroxy propoxy phenyl)} methane, four diethylene glycols, and dipropylene glycol, are mentioned.

[0011] The polyether diol which adds a carbon number 2 or the alkylene oxide of 3 to aliphatic series polyalkylene glycols, such as one polyethylene glycol, a polypropylene glycol, and a polytetramethylene glycol, the aliphatic series glycol which carried out 2 above, an alicycle group glycol, or an aromatic series glycol, and is obtained as polyether diol which will constitute the polyether ester of A-1, and the polyether diol which adds a carbon number 2 or the alkylene oxide of 3 to 3 aliphatic-series primary amine, and is obtained are mentioned. This polyether diol has the desirable thing of molecular weight 400-6000, and its thing of 1000-4000 is still more desirable.

[0012] the polyether ester of A-1 -- said aromatic series or saturation aliphatic series dicarboxylic acid carried out -- more than 85 mol % -- it is obtained by the condensation polymerization of the included dicarboxylic acid or its ester plasticity derivative, said glycol carried out, and said polyether diol carried out. In this case, as dicarboxylic acid, it is less than [ 15 mol % ], and the dicarboxylic acid which has an ionicity radical at 3-13-mol % preferably can be used. Although what has sulfonic groups, such as 5-sulfoisophtharate sodium, sulfo terephthalic-acid sodium, 4-sodium sulfo naphthalene -2, and 6-dicarboxylic acid, is mentioned as dicarboxylic acid which has this ionicity radical, 5-sulfoisophtharate sodium is advantageous especially.

[0013] In the polyether ester of A-1, although especially the configuration rate of the above mentioned glycol and the above mentioned polyether diol is not limited, it is desirable to be referred to as a glycol / polyether diol =90 / 10 - 50/50 (mole ratio), and it is still more desirable to be referred to as 85 / 15 - 60/40 (mole ratio). Moreover, in the polyether ester of A-1, it is desirable to make the configuration rate of the block part by polyether diol into 50 - 95% of the weight of the whole, and it is still more desirable

to consider as 60 - 90 % of the weight. And although the polyether ester of A-1 uses the thing of average molecular weight 1000-100000, it is usually desirable to use the thing of 2000-15000.

[0014] As dicarboxylic acid of the aromatic series which will constitute the polyether ester amides of A-2, or saturation aliphatic series, what was described above about A-1 can be mentioned. In this case, aromatic series dicarboxylic acid / aliphatic series dicarboxylic acid = it is desirable to be referred to as 100 / 0 - 15/85 (mole ratio), and it is still more desirable to be referred to as 100 / 0 - 30/70 (mole ratio).

[0015] The amide plasticity compound which will constitute the polyether ester amides of A-2 is chosen from diamine, a lactam, and an amino carboxylic acid. As this amide plasticity compound, one hexamethylenediamine, Aliphatic series diamine [ , such as nonamethylene diamine, ], 2,4, and 4'-diamino diphenyl ether, - diamino diphenylmethane, and 4 and 4 '4, 4'-diaminodiphenyl sulfone, Although amino alkane carboxylic acids, such as lactams, such as aromatic series diamines, such as a 4 and 4'-diamino diphenyl sulfide, 3 epsilon caprolactam, and omega-RAURO lactam, a 411-amino undecane carboxylic acid, and 6-amino hexane carboxylic acid, are mentioned A hexamethylenediamine and epsilon caprolactam are advantageous especially.

[0016] As polyether diol which will constitute the polyether ester amides of A-2, what was described above about A-1 can be mentioned. In this case, as polyether diol, the thing of molecular weight 400-6000 is desirable, and the thing of 1000-4000 is still more desirable.

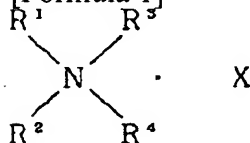
[0017] the polyether ester amides of A-2 -- said aromatic series or aliphatic series dicarboxylic acid carried out -- more than 90 mol % -- it is obtained by the reaction of the included dicarboxylic acid or its ester plasticity derivative, an amide plasticity compound, and polyether diol. In this case, as dicarboxylic acid, the dicarboxylic acid which has an ionicity radical in not more than 10 mol % can be used. As dicarboxylic acid which has this ionicity radical, what was described above about A-1 is mentioned.

[0018] In the polyether ester amides of A-2, although especially the configuration rate of the above mentioned amide plasticity compound and the above mentioned polyether diol is not restricted, it is desirable to be referred to as an amide plasticity compound / polyether diol = 20 / 80 - 80/20 (mole ratio), and it is still more desirable to be referred to as 35 / 65 - 65/35 (mole ratio). Moreover, in the polyether ester amides of A-2, it is desirable to make the configuration rate of the block part by polyether diol into 50 - 95% of the weight of the whole, and it is still more desirable to consider as 60 - 90 % of the weight. And although the polyether ester amides of A-2 usually use the thing of average molecular weight 1000-100000, it is desirable to use the thing of 2000-20000, and it is still more desirable to use the thing of 5000-15000.

[0019] The nitrogen-containing compound of B has one or more ammonium and the hydrocarbon group of two or more carbon numbers [ 12 or more ] in intramolecular. As this nitrogen-containing compound, the compound shown by the following formula 1, the acid neutralization object of the compound shown by the following formula 2, the compound which carried out condensation generation of at least two mols or more using the divalent reactant compound of the compound shown by the following formula 2, or its salt is mentioned.

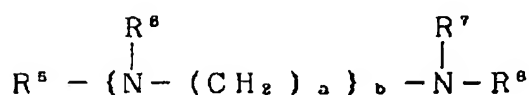
[0020]

[Formula 1]



[0021] The hydroxy (Pori) alkoxyalkyl group R3 which has the alkanoyl aminopropyl radical which has an alkanoyl aminoethyl radical and the alkanoyl radical of carbon numbers 12-22 which have R1, the aliphatic-hydrocarbon radical of the R2:carbon numbers 12-22, and the alkanoyl radical of carbon numbers 12-22, a carbon number 2 or the alkoxy group of 3, and an alkyl group in a formula 1, R4:hydrogen, the alkyl group of carbon numbers 1-4, or the aralkyl radical X: Anionic radical [0022]

[Formula 2]



[0023] a formula 2 -- setting -- R5-R8: -- at least two pieces -- the alkanoyl radical of carbon numbers 12-22, or an ARUKE noil radical -- it is -- the remainder -- hydrogen or the alkyl group of carbon numbers 1-4 -- it is -- and the 1st class of at least one isolation of the amino group or the integer of integer b:2-4 of 2 class amino-group a:3-5 [0024] As a compound shown by the formula 1, 1 dilauryl dimethylammonium chloride, 1 long-chain alkyl (or alkenyl) ammonium compounds, such as distearyldimethylbenzylammonium methylsulfonate, 2) Dilauryl monomethylamine and acetate, a distearyl amine, a p-toluenesulfonic-acid salt, etc., The inorganic acid of a 1 long-chain alkyl (or alkenyl) amine, or the salt of an organic acid, 3) The salt of 1 (N-diacyl amino alkyl) amines, such as a diethylenetriamine distearyl amide and acetate, and a dipropylenetriamine distearyl amide, acetate, the acetate of the ethylene oxide addition product of a diethylenetriamine distearyl amide, etc. are mentioned.

[0025] As an acid neutralization object of the compound shown by the formula 2, the neutralization object of partial acylation polyalkylene polyamine and acids, such as a triethylenetetramine distearyl amide octyl phosphate salt, and a tetraethylenepentamine distearyl amide, acetate, is mentioned.

[0026] What is necessary is to react with the amino group of the 1st class or isolation of the 2nd class, and just to carry out condensation generation of at least two mols or more of the compound shown by the formula 2 as a divalent reactant compound which carries out condensation generation of at least two mols or more of the compound shown by the formula 2. As this divalent reactant compound, alkylene carbonate compounds, such as diisocyanate compounds, such as diepoxy compounds, such as 1 ethylene glycol diglycidyl ether, diethylene-glycol diglycidyl ether, and polyethylene glycol diglycidyl ether, 2 tolylene diisocyanate, and methylenebis phenyl isocyanate, 3 ethylene carbonate, and propylene carbonate, 4 epichlorohydrin, a urea, etc. are mentioned.

[0027] Although what carried out condensation generation using a divalent reactant compound which described above at least two mols or more of the compound shown by the formula 2 can also be used as it is, the thing which neutralized these using the inorganic acid or the organic acid further, or the thing which used the third class amino group as quarternary ammonium salt using the proper fourth class-ized reagent can also be used. Also in these, it is advantageous as a nitrogen-containing compound of B to use the neutralization object of partial acylation polyalkylene polyamine and an acid.

[0028] In this invention, a hydrophobic synthetic fiber is processed using the polyether ester of A and/or polyether ester amides, and the nitrogen-containing compound of B. In this case, the operating rate of A and B is set to A/B=95 / 5 - 50/50 (weight ratio), and is preferably set to 90 / 10 - 55/45 (weight ratio). It is for acquiring a desired hydrophilic property and its endurance.

[0029] Moreover, in this invention, A and B are used so that such sum total coating weight may become 0.2 - 1.5 % of the weight preferably 0.1 to 3% of the weight to a hydrophobic synthetic fiber. When sum total coating weight is less than 0.1%, suitable effectiveness is not acquired, even if it is difficult to acquire the stable effectiveness and it exceeds 3 % of the weight conversely.

[0030] It is desirable to use the polyether ester of A and/or polyether ester amides as a water dispersion or a water solution on the occasion of operation of this invention. When preparing a water dispersion, a dispersant can be used auxiliary. As this dispersant, anionic surface active agents, such as nonionic surface active agents, such as 1 polyoxyethylene (it is hereafter written as POE) alkyl ether, the POE alkylphenyl ether, and POE alkyl ester, 2 alkyl sulfate, POE alkyl sulfate, alkyl sulfonate, alkylbenzene sulfonate, alkyl phosphate, and a POE alkyl phosphate salt, etc. are mentioned. As for the operating rate of these dispersants, it is desirable to consider as 15 or less % of the weight to A. When preparing the water dispersion or water solution of A, an organic solvent can also be suitably used together. Similarly, it can use as a water dispersion or a water solution also about the nitrogen-containing compound of B. When preparing a water dispersion or a water solution, as solid content concentration, it considers as 3 - 15 % of the weight preferably, and this can usually be used one to 20% of the weight, diluting it with

water further.

[0031] In this invention, the polyether ester of A and/or polyether ester amides, and the nitrogen-containing compound of B may process a hydrophobic synthetic fiber, using these independently, and may process a hydrophobic synthetic fiber, using these simultaneously. When using these independently, after processing using A, B can also be used and processed, but after processing using B first, it is desirable to use and process A next through a desiccation process.

[0032] This invention is applicable at the production process and processing process of a hydrophobic synthetic fiber. It can apply to the raw thread which passed through the spinning process and drawing process of a hydrophobic synthetic fiber, and can also apply, post processing, for example, the spinning process etc., of raw thread etc. As the application approach, dip coating, a spray method, the roller supplying [ with oil ] method, etc. are mentioned.

[0033] The bicomponent fiber which used these two or more sorts else [ , such as polyester fiber, a polyamide fiber, polyacrylonitrile fiber, and a polypropylene fiber, ], and was made into the fiber of various forms as a hydrophobic synthetic fiber which can apply this invention is mentioned. The manifestation of the effectiveness at the time of applying this invention of polyester fiber is remarkable especially. There are the refining polyester fiber which copolymerized various monomers for polyester fiber like polyethylene terephthalate polyethylene isophthalate and polyethylene terephthalate polybutylene terephthalate and the various objects else [ , such as polyethylene terephthalate, polybutylene terephthalate, and polyethylenenaphthalate, ], for example, basicity, acid tingibility polyester fiber, antistatic nature polyester fiber, fire-resistant polyester fiber, etc. as this polyester fiber.

[0034]

[Example]

Test partition 1 (composition of A)

- the synthetic dimethyl terephthalate 91 section (the weight section --) of polyether ester (A-1a) Below The isophthalic acid dimethyl 10 same section, the ethylene glycol 68 section, As the polyethylene-glycol 403 section of a mean molecular weight 3100, and a catalyst, the manganese acetate 4 monohydrate 0.15 section, Teach the antimony-trioxide 0.05 section and an ester interchange is performed at 150-230 degrees C to the bottom of a nitrogen gas air current. The phosphorous acid 0.035 section was added after distilling off the methanol of the specified quantity, and it was gradually made reduced pressure, requiring and carrying out temperature up of the 2 hours at 270 degrees C, and the polycondensation was eventually performed for 40 minutes in 270 degrees C under reduced pressure of 1mmHg. The average molecular weight of the polystyrene conversion by GPC of the obtained polyether ester (A-1a) was 9500. Polyether ester (A-1b, A-1c, A-1d) was compounded like the following. Those contents were shown in a table 1.

[0035] - The manganese acetate 4 monohydrate 0.10 section and the antimony-trioxide 0.03 section were taught as the synthetic dimethyl terephthalate 58 section of a polyether ester amide (A-2a), the isophthalic acid dimethyl 10 section, the polyethylene-glycol 525 section of a mean molecular weight 1500, the epsilon caprolactam 34 section, and a catalyst, it heated at 160-230 degrees C under the nitrogen gas air current, the phosphorous acid 0.024 section was added after distilling off the methanol of the specified quantity, and the polycondensation was performed for 60 minutes in 270 degrees C. The average molecular weight of the polystyrene conversion by GPC of the obtained polyether ester amide (A-2a) was 7500. The polyether ester amide (A-2b, A-2c) was compounded like the following. Those results were shown in a table 1.

[0036]

[A table 1]



区 分		A - 1 相当				A - 2 相当		
		A-1a	A-1b	A-1c	A-1d	A-2a	A-2b	A-2c
1)	1)-1	45	35	15	15	30	18	30
	1)-2	5	10	35		5	2	
	1)-3				30		30	
	1)-4		5		5			5
2)	2)-1	37	37	35	35			
	2)-2			5				
3)	3)-1					35		35
	3)-2	13					20	
	3)-3		13					
	3)-4			10				
	3)-5				15			
4)	4)-1						30	
	4)-2					30		30
平均分子量		9500	8500	11000	9000	7500	8000	7000
*1		82	83	81	63	88	87	88

[0037] In a table 1 among the table of the column of 1-4 Numerical: mol % 1: dicarboxylic acid, However, a 1-1; terephthalic acid, 1-2; isophthalic acid, 1-3; An adipic acid, 1) -4; 5-sulfoisophtharic acid sodium 2: -- a glycol -- however 2) -1; -- ethylene glycol and 2-2; 1,4-butanediol 3: polyether diol -- However, the polyethylene glycol of the 3-1; mean molecular weight 1500, 3-2; The polyethylene glycol of a mean molecular weight 3100, 3) -3; -- the cyclohexane dimethanol ethyleneoxide addition product of a mean molecular weight 3600 -- 3) -4; -- the polyoxyethylene lauryl amino ether of a mean molecular weight 4000 -- 3) -5; -- the bisphenol A ethyleneoxide addition product 4: amide plasticity compound of a mean molecular weight 1100 however a 4-1; hexamethylenediamine, and a 4-2; epsilon caprolactam

\*1: polyalkylene glycol content (% of the weight)

[0038] Test partition 2 (processing and its assessment of a hydrophobic synthetic fiber)

- The water dispersion of 10 % of the weight of solid content concentration which has the presentation of a publication in one to examples 1-12 and example of comparison 11 table 2 and a table 3 was prepared. Water dilution of this was carried out further, and it considered as 1 % of the weight of solid content concentration. It was immersed in the water dispersion which carried out water dilution of the polyester staple cotton (2.0 denier x 50mm, cleaning article) by the above with the bath ratio 30:1, and the sample cotton made into 0.3 % of the weight of target coating weight of solid content was obtained as 30% of contraction percentages. The following assessment was presented without drying this sample cotton. The result was shown in a table 4.

[0039] .. After putting 70ml (25 degrees C) of assessment water of initial dispersibility into a test tube with a diameter [ of 2.8cm ] x height of 20cm, dropping 0.1g of sample cotton on this quietly and making a plug a test tube, it was quietly made reversed twice. By viewing, the distributed condition of the cotton of 10 minutes after was judged on the following criteria.

[0040] .. criterion O; of primary stage dispersibility -- O; in which it sediments altogether and condensation fiber is not accepted at all -- [0041] with it [ much / x; floating fiber or condensation fiber a part of \*\*; floating fiber condensation fiber is slightly accepted to be is accepted to be, or condensation fiber is clearly accepted to be is remarkable, and / although it sedimented altogether ] .. After the assessment above of endurance estimated initial dispersibility, the test tube was shaken for 1 minute with the paint shaker (Oriental energy machine factory company make). By viewing, the cellular adhesion to the cotton of 10 minutes after and the amount of sedimentation of cotton were judged on the following criteria.

[0042] .. criterion O; of cellular adhesion -- O; in which adhesion of air bubbles is not accepted at all -- \*\*; in which adhesion of air bubbles is accepted slightly -- [0043] adhesion of air bubbles is accepted to be to the whole x; fiber adhesion of air bubbles is accepted to be to a part .. criterion O; of the amount of sedimentation -- O; to which all fiber sedimented -- \*\*; which has a floating fiber very slightly -- [0044] to which the fiber more than x; one half where a part of fiber is floating is floating

[A table 2]

区 分		実 施 例											
		1	2	3	4	5	6	7	8	9	10	11	12
A	A-1a	63							50		50		72
	A-1b		72							33		72	
	A-1c			82									
	A-1d				63								
	A-2a					63			13				
	A-2b						63			30			
	A-2c							63					
	D-1	7	8	5					7	7	5	8	8
	D-2				7	7	7	7					
B	B-1	30	20		30	30		30	30		35	20	20
	B-2			13						30			
	B-3						30						
その他	E												
	F												
	G												
	H										10		
A/B(重量比)		68/32	78/22	86/14	68/32	68/32	68/32	68/32	68/32	68/32	59/41	78/22	78/22

[0045]

[A table 3]

区 分		比 較 例										
		1	2	3	4	5	6	7	8	9	10	11
A	A-1a	90								63		
	A-1b		90								72	72
	A-1c						27					
	A-1d							9				
	A-2a			90								
	A-2b								13.5			
	A-2c											
	D-1	10	10	10			3	1	1.5	7	8	8
	D-2											
B	B-1				100			90				
	B-2						70					
	B-3					100			85			
その他	E									30		
	F										20	
	G											20
	H											
A/B(重量比)		100/0	100/0	100/0	100/100	100/100	29/71	9/91	14/86	100/0	100/0	100/0

[0046] In a table 2 and a table 3 among A, B, and the table of other columns The acetate of the ethyleneoxide (five mols) addition product of a thing B-1; diethylenetriamine distearyl amide given in the numerical:% of the weight A-1 a-A-2c; table 1, B-2; triethylenetetramine distearyl amide acetate, B-3; acetate D-1 of the reactant of a diethylenetriamine distearyl amide (two mols) and epichlorohydrin (one mol) ;P OE (six mols) nonylphenyl ethersulfate ammonium, D-2 ;P OE (four mols) laurylether sulfate triethanolamine E; lauroyl aminopropyl trimethylammonium chloride, F; stearyl amine ethyleneoxide (12 mols) addition product, G; distearyl phosphate Na salt [0047]

[A table 4]

区 分		実 施 例											比 較 例										
		1	2	3	4	5	6	7	8	9	10	11	1	2	3	4	5	6	7	8	9	10	11
初期分散性		◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	△	△	△	×	×	×	×	×	△	△	△
耐久性	気泡付着性	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	×	×	×	×	×	×	×	×	×	△	△
	沈 降 量	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	◎	×	×	×	×	×	×	×	×	△	△	×

[0048] Test partition 3 (preparation of the water dispersion of A and/or B)

- Although preparation polyether ester (A-1a) 90g of the water dispersion of polyether ester (A-1a) was changed into the 170-180-degree C melting condition and polyoxyethylene (six mols) nonylphenyl ethersulfate ammonium 10g was beforehand dissolved in 900g of 30-degree C water as a dispersant, it added to inside, agitating by the homomixer, and the water dispersion was prepared to it. This water dispersion contains a dispersant for polyether ester (A-1a) 1% of the weight 9% of the weight. Similarly, the water dispersion which contains a dispersant for polyether ester (A-1b) 1% of the weight 9% of the weight was prepared.

[0049] - Using preparation polyether ester amide (A-2a) 90g of the water dispersion of a polyether ester amide (A-2a), polyoxyethylene (six mols) nonylphenyl ethersulfate ammonium 10g, and 900g of water, it was operated like the case of preparation of the water dispersion of polyether ester (A-1a), and the water dispersion which contains a dispersant for a polyether ester amide (A-2a) 1% of the weight 9% of

the weight was prepared. Similarly, the water dispersion which contains a dispersant for a polyether ester amide (A-2b) 1% of the weight 9% of the weight was prepared.

[0050] - Heating fusion of the mixture (preparation polyether ester (A-1a) 45g of the mixed water dispersion of polyether ester (A-1a) and a polyether ester amide (A-2b) and polyether ester amide (A-2b) 45g) was carried out at 170-180 degrees C. It added by having made this into the dispersant beforehand, agitating 10g of polyoxyethylene (six mols) lauryl ether sulfate triethanolamine salts by the homomixer into the water solution which dissolved in 900g of 30-degree C water, and the mixed water dispersion was prepared. This mixed water dispersion contains [ polyether ester (A-1a) ] 4.5 % of the weight and a dispersant for a polyether ester amide (A-2b) 1% of the weight 4.5% of the weight. Similarly, the mixed water dispersion which contains [ polyether ester (A-1b) ] 4.5 % of the weight and a dispersant for a polyether ester amide (A-2b) 1% of the weight 4.5% of the weight was prepared.

[0051] Test partition 4 (preparation of spray liquid, processing of polyester staple cotton, and assessment)

- The water dispersion and the mixed water dispersion which were obtained in the preparation test partition 3 of the 2nd liquid (spray liquid containing A) were diluted with water, and the 2nd liquid which has the solid content concentration of a publication in a table 5 and a table 6 was prepared.

[0052] - The 1st liquid which has the solid content concentration of a publication at a table 5 and a table 6 using the nitrogen-containing compound of a publication in the preparation table 5 and table 6 of the 1st liquid (spray liquid containing B) was prepared.

[0053] - To the polyester staple cotton used in examples 13-20 and the example 12 of a comparison - 17 test partitions 2, spray oiling was carried out so that it might become a table 5 and a table 6 with 30 % of the weight of wet pickup about the 1st liquid of a publication. Succeedingly, spray oiling of the 2nd liquid given in a table 5 and a table 6 was carried out so that it might become with 30 % of the weight of wet pickup, and the sample cotton of a publication was obtained to a table 5 and a table 6. About the sample cotton obtained here, it was similarly estimated as the test partition 2. The result was shown in a table 5 and a table 6.

[0054]

[A table 5]

区 分	実 施 例							
	13	14	15	16	17	18	19	20
(第2液)					A-1a	A-1b		
種類	A-1a	A-1b	A-2a	A-2b	A-2b	A-2b	A-1a	A-1a
濃度(重量%)	0.8	0.9	0.9	0.8	0.8	0.95	2.0	4.0
(第1液)								
種類	B-1	B-3	B-1	B-1	B-1	B-3	B-1	B-1
濃度(重量%)	0.3	0.2	0.2	0.3	0.3	0.15	0.8	1.5
A/B(重量比)	68/32	80/20	80/20	68/32	68/32	85/15	69/31	78/22
A+B付着量(重量%)	0.31	0.30	0.30	0.31	0.31	0.30	0.78	1.53
初期分散性	◎	◎	◎	○	◎	◎	◎	◎
耐久性	◎	◎	◎	◎	○	◎	◎	◎
沈 降 量	◎	◎	○	○	◎	◎	◎	◎

[0055]

[A table 6]

区 分	比較 例					
	12	13	14	15	16	17
(第2液)					A-1b	A-1b
種類	A-1a	A-1a	A-2a	A-2a	A-2b	A-2b
濃度(重量%)	0.12	10.0	0.16	10.0	0.16	10.0
(第1液)						
種類	B-1	B-1	B-1	B-1	B-3	B-3
濃度(重量%)	0.05	4.2	0.04	2.3	0.07	4.2
A/B(重量比)	70/30	68/32	60/20	60/20	68/32	68/32
A+B付着量(重量%)	0.05	4.0	0.06	3.4	0.06	4.0
初期分散性	×	○	×	○	×	○
耐久性	×	×	×	△	×	×
気泡付着性	×	×	×	△	×	×
沈 降 量	×	△	×	△	×	△

[0056] - According to the "hand-made paper adjustment approach" of JIS-P8209, sample paper was produced using the sample cotton obtained in examples 21 and 22 and the example 18 of a comparison, 19 examples 1 and the example 14, the example 1 of a comparison, and the example 15 of a comparison. The tensile strength by JIS-P8113 and the formation by viewing were measured about the sample paper obtained here. The result was shown in a table 7.

[0057]

[A table 7]

区 分	試料綿	引張強さ (kg/15mm)	地 合
実施例21	実施例 1	0.41	良好
実施例22	実施例14	0.38	良好
比較例18	比較例 1	0.26	ムラ有り
比較例19	比較例15	0.29	ムラ有り

[0058]

[Effect of the Invention] There is already effectiveness that advanced first stage water-dispersion and its outstanding endurance can be given to a hydrophobic synthetic fiber in this invention explained above so that clearly.

[Translation done.]